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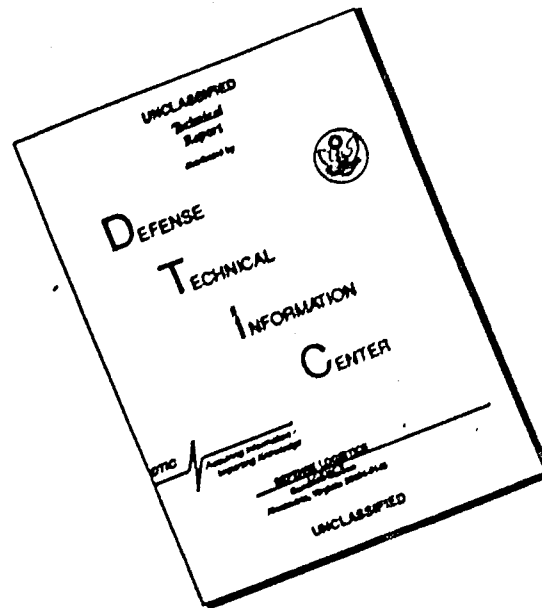
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QUARTERLY PERIODIC STATUS REPORT
OF THE
HYDROGEN PEROXIDE LABORATORIES

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I. Stability of Hydrogen Peroxide (C. C. Lane and H. L. Kimball)

A. The Effect of Aluminum Upon Stabilization with Tin

The principal subject of investigation, during the present quarter, carried out by the stability group of the hydrogen peroxide project, was concerned with the desire to determine the reason underlying the observed decrease with time in the stabilizing effect of sodium stannate when added to concentrated hydrogen peroxide in aluminum containers. For example, in a particular sample of 90% hydrogen peroxide with a tin content of 30 p.p.m. Sn (added as $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$), two days after the hydrogen peroxide solution was placed in an aluminum container the tin content was observed to have dropped to 20 p.p.m. Sn. Further decline in tin content proceeded at a progressively slower pace.

The following series of tests was undertaken in an attempt to establish, if possible, a relationship between the rate of decomposition of the hydrogen peroxide solution and its content of aluminum ion, tin (as stannate), and known added contaminants, such as ferric ion. In each case the rate of decomposition at 50°C was determined. The pH of the solutions was taken but was not adjusted to constancy, and some influence on the observed rates of decomposition is therefore expected, due to variation in the respective pH's in the several tests. In all cases the starting material for the tests was 90% "Becco" hydrogen peroxide with no intentionally added stabilizer. In those cases where iron contamination was sought, 0.1 p.p.m. of Fe^{+++} ion was employed, by addition of the hydrated sulfate.

The hydrogen peroxide solutions were contained in 250-ml. conditioned pyrex flasks and were held at 50°C by immersion in a constant temperature bath. The period of test was on the average nine days. The base measurement on the 90% H_2O_2 alone was observed to be 0.0026 percent decomposition per hr. at 50°C .

The additives employed in the tests included:

Test

- (A) 24 p.p.m. Sn^{4+} (added as $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$).
- (B) 24 p.p.m. Sn^{4+} + 0.1 p.p.m. Fe^{+++} (added as $\text{Fe}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$).
- (C) 30 p.p.m. Al^{+++} added as $\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$ + 24 p.p.m. Sn^{4+} + 0.1 p.p.m. Fe^{+++} .
- (D) 15 p.p.m. Al^{+++} + 24 p.p.m. Sn^{4+} + 0.1 p.p.m. Fe^{+++} (added after 1 day).
- (E) 30 p.p.m. Al^{+++} .
- (F) Same as (C) but Fe^{+++} added only after 8 days.
- (G) 30 p.p.m. Al^{+++} + 0.1 p.p.m. Fe^{+++} .

The rates of decomposition observed with the solutions containing the above mixtures are plotted in Figure 1.

Results:- As was expected, the lowest rate of decomposition was obtained from the solution containing 24 p.p.m. Sn^{4+} alone in the 90% hydrogen peroxide solution. (Test A); the observed rate being 0.0008% per hr. at 50°C . (see Curve A in Fig. 1).

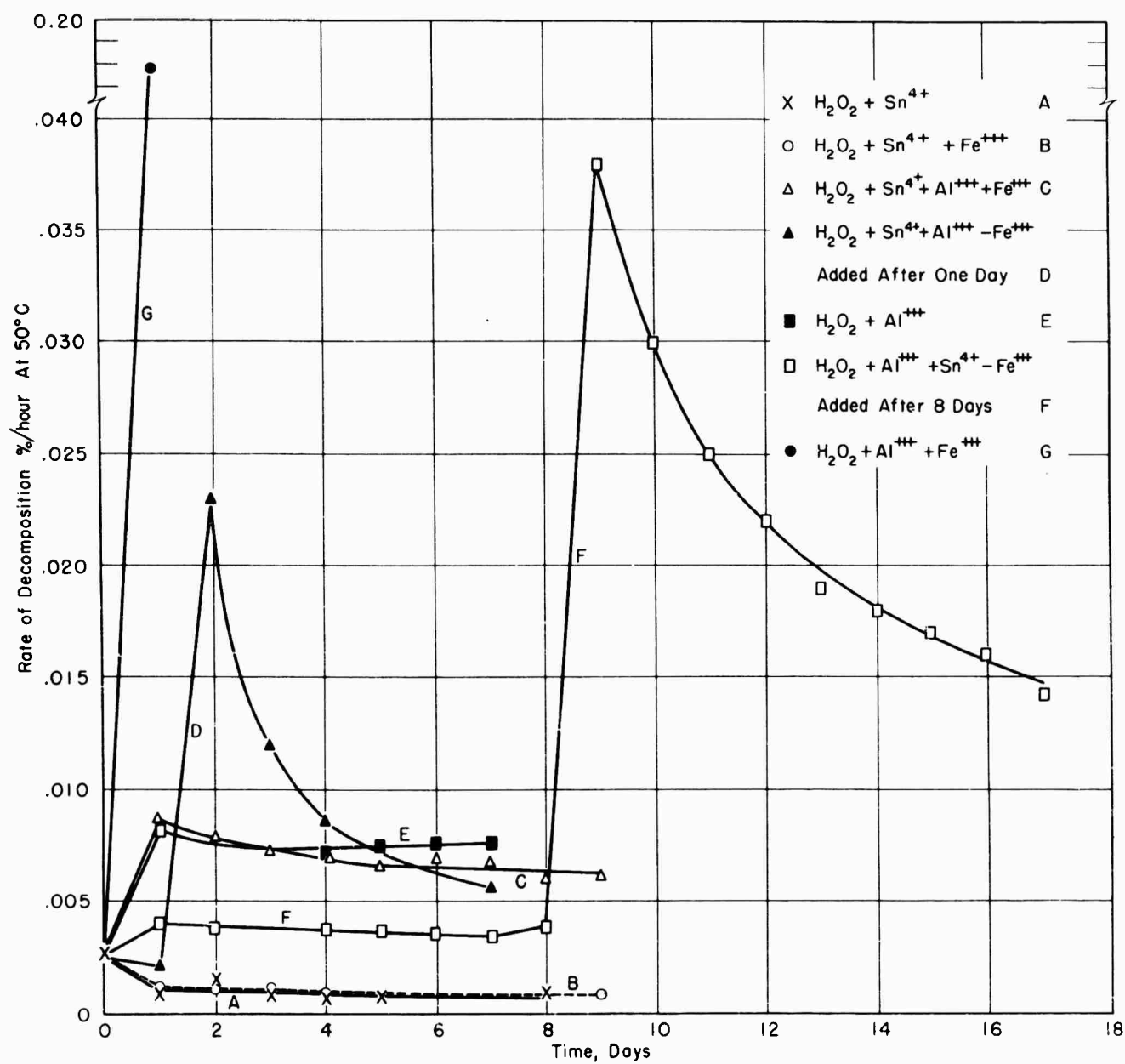


FIG 1- RATE OF DECOMPOSITION OF 90% H_2O_2 CONTAINING TIN, ALUMINUM, AND IRON

Addition of aluminum ion alone (added as hydrated sulfate) resulted in a rise in the decomposition rate to about 0.0075% per hr. (Curve E). It is conceivable that this degree of increase in the rate was due to the small amount of iron contamination which the aluminum sulfate crystals were known to contain.

The addition of 30 p.p.m. of stannic tin (added as stannate) as well as of the 30 p.p.m. of aluminum sulfate caused a reduction in the rate from 0.0075%, shown by the aluminum alone, to an average of 0.0037% per hr. (see curve F up to 8 days). On expressing it in another way, the decomposition rate observed was increased to about four times that found for the stannate alone (Test A) by addition of the aluminum solution.

Curve B, which is very nearly coincident with curve A over a week's time, indicates that in the presence of 24 p.p.m. of tin (added as stannate) sufficient protection was afforded so that 0.1 p.p.m. of Fe^{+++} contamination was held completely in check. However, in the absence of the stannate the same amount of iron contamination, together with the effect of 30 p.p.m. of added aluminum ion, caused a very sharp rise in the decomposition rate of the peroxide solution (curve G). This rise is mainly due to the iron contamination, as addition of aluminum ion alone above referred to (curve E) had a relatively small effect in the same concentration (30 p.p.m.).

The same sharp rise in the decomposition rate is seen in Curve D, which applies to the test in which, following addition of 15 p.p.m. Al^{+++} (added as sulfate), 0.1 p.p.m. of Fe^{+++} was introduced after the lapse of one day. Again in a comparison of curves C and F, both of which were concerned with aluminum ion and stannate addition, the effect of addition of 0.1 p.p.m. of ferric ion (in curve F) is seen to be pronounced when it was withheld for 8 days. After this period, added iron caused a sharp increase in the decomposition rate of the peroxide.

Since the effect of variation in pH of the solution is well known, readings were taken at the completion of the tests with the Beckman Type G meter, at tenfold dilution of the peroxide, throughout the series of tests. The observed values follow:-

Test	pH reading (1:10) dilution
A	5.90
B	5.81
C	4.08
D	*
E	4.43
F	3.10
G	4.30
* Run incomplete	

Conclusions:- The fact that the addition of iron caused a more accentuated rise in the decomposition rate of the hydrogen peroxide when such addition was withheld for 8 days appears to indicate that the protective action of the stannate had declined appreciably in the same period, so that when the iron was added the hydrogen peroxide solution was ineffectively stabilized and a sharp increase in the decomposition rate ensued.

Furthermore, it is noted that the sharp rise in decomposition rate caused by ferric ion addition is in each case quickly followed by an abrupt decline in the rate. It seems reasonable to attribute this decline to a coagulation of the catalytically active hydrous ferric oxide (or basic ferric salt) which is first formed by hydrolysis of the added ferric sulfate. The consequent diminution in the surface of the active colloidal material results in a decrease in the observed rate of decomposition of the peroxide.

Although the experiments thus far reported appear to indicate a slight accelerative effect of aluminum upon the decomposition rate of hydrogen peroxide, further tests are being currently conducted in which metallic aluminum is being introduced into the solution, in the form of strips of the metal, to observe the influence of the metal upon the stannate-stabilized peroxide. It should be possible to determine whether or not metallic tin is displaced by the aluminum in such solutions; if so, the stabilizing effect of the original stannate (or hydrous stannic oxide derived therefrom) will be expected to be lacking and added catalytic ions (such as ferric ion) should bring about a pronounced rise in the decomposition rate of the peroxide solution. It is hoped also that it may also be possible to follow the course of these phenomena with the help of the conductivities of the solution, as measured with a conductivity cell with tin electrodes.

B. Stability Measurements on Shell 90% Hydrogen Peroxide

Two 24-oz. bottles of 90% hydrogen peroxide, produced by the Shell Development Company process, were received during this quarter. The analyses and decomposition rate of the solution as reported by the company and as determined in our laboratory are as follows:

	<u>Shell Analysis</u>	<u>Our Data</u>
H ₂ O ₂ Cono.	91.5%	90.2%
pH (1:10 dilution)	4.2	4.58
Decomp. Rate	0.29%/24 hr./100°C	0.00136%/hr/50°C.
Carbon %/wt.	0.007	
Residue mg/l.	3.3	
Phosphate (mg/l.)	0.2	
Sulphate "	1.0	
Tin "	0.4	

	<u>Shell Analysis</u>	<u>Our Data</u>
Aluminum (mg/l.)	0.02	
Nitrate "	2.5	

This sample of 90% unstabilized peroxide is evidently of high quality: its decomposition rate compares favorably with the best we have examined. It is planned to dilute this material with carefully purified water and to determine the stabilities of these diluted solutions. Further checks on the analysis of the solution, as received, are also planned.

II. Flame Velocities in Hydrogen Peroxide Vapor (M.A.T. Mendes)

In proceeding with the experiments to determine the rate of decomposition of hydrogen peroxide vapor over hot liquid hydrogen peroxide, several modifications have been made in the apparatus described in the previous quarterly report. A glass well for a thermocouple (iron-constantan) together with a two-way stopcock have been connected to the bottom of the tube (31 cm long, 1.755 cm diameter) containing the hydrogen peroxide. One of the stopcock outlets has been used to remove samples of hydrogen peroxide--one before igniting and the other after some 15 velocity measurements had been taken. Once the second sample had been obtained the decomposition was terminated by flooding with water injected through the other inlet of the stopcock.

An improvement was also made in the heating system so that a uniform temperature was obtained throughout the liquid without manual stirring. A hot platinum wire is now being used to ignite the hydrogen peroxide vapor instead of the gas flame used previously. The wire is heated and cleaned with concentrated nitric acid prior to starting the flame, in order to avoid any contamination of the liquid. Slight amounts of impurities present were found to be responsible, not only for an increase in the liquid temperature but also in the amount of decomposition of the liquid.

The initial experiments were made with 99 weight per cent hydrogen peroxide (Becco) and runs have now been made covering a wider range of concentrations in order to obtain quantitative information on the effect of concentration on the flame velocity.

Table I summarizes the data obtained to date. Figure 2 is a plot of the variation of the burning velocity data with H_2O_2 concentration. The effect, if any, of liquid temperature on burning velocity is still uncertain. Actually the temperature range over which flames can be sustained is quite small since it must be above the temperature at which a flame can be ignited and below the liquid boiling temperature. In any event, it appears that the effect of liquid concentration on burning velocity is far more important than that of liquid temperature.

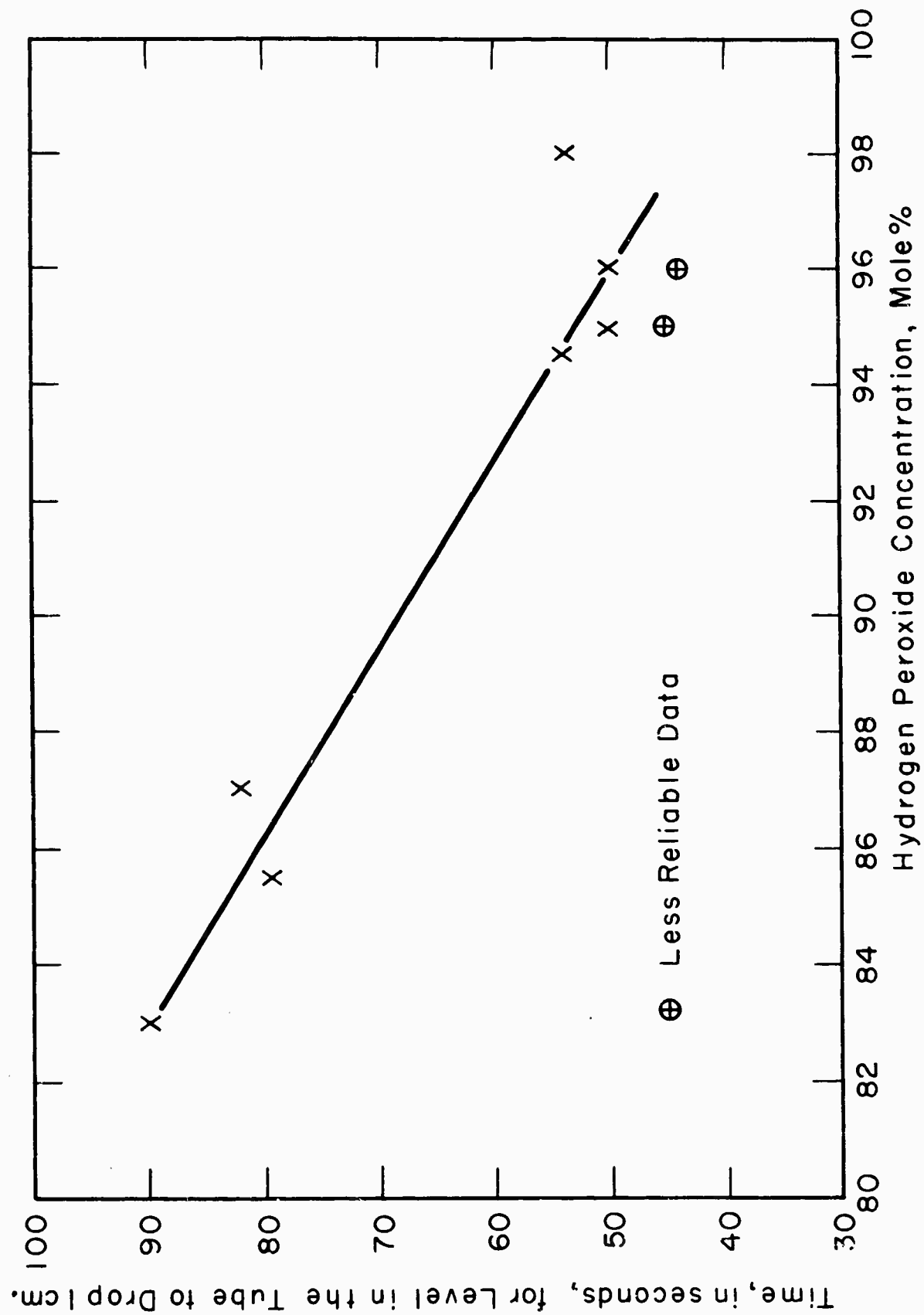


FIG 2—EFFECT OF CONCENTRATION UPON RATE OF CONSUMPTION OF
 H_2O_2 BY DECOMPOSITION OF VAPOR OVER LIQUID

Table I

Run No.	Conc ¹ mole %	Liquid Consumption Rate (sec)*	Liq. Temp. °C	Temp of °C**	Flame Veloc. ft/sec.***
6	96	43-45	122	-	-
7	95	50	117	-	0.84
11	95	44-46	120	723	0.92
12	94.5	53-55	117	640	0.79
13	96	49-51	110-114	855	0.82
14	98	52-55	110-114	547	0.77
15	87	81-83	110	607	0.51
17	85.5	78-80	112	657	0.53
18	83	89-91	114	632	0.47

1 Average of liquid composition at beginning and end of run.

* Time, in seconds for the liquid level to drop 1 cm. in the tube.

** As measured by an iron-constantan thermocouple placed in the product gases about 5 cm. above the liquid surface.

*** See text for explanation.

The effect of the flame on the decomposition rate of the liquid-phase hydrogen peroxide still remains to be studied. It has been noticed that the hydrogen peroxide decomposes at a greater rate when the flame is present than by the simple heating effect of the liquid at the temperature of the experiment.

In respect to this observation an attempt will be made to determine the influence of the flame temperature on the decrease in hydrogen peroxide concentration during a run.

At the same time an attempt will be made to correlate the flame velocity data obtained for the experiments described here with those previously reported for the vapor phase by the bunsen burner technique. In Table I are included some "flame velocities" computed from the data obtained by observation of the rate of liquid consumption. These flame velocities are the rate at which vapor at saturation temperature is consumed if it is assumed that reaction takes place on a horizontal flame front of area equal to the cross-section of the glass tube holding the liquid. As such these numbers are probably of interest only as an order of magnitude.

III. Supercooling of Liquid Hydrogen Peroxide (J. Roberts)

All of the experiments reported here regarding the supercooling of liquid hydrogen peroxide have been carried out in a 13 m.m. Pyrex test tube fitted with a 1 1/2 in. long, 3 mm diameter Pyrex tube inserted through the base. A thermocouple lead was placed in the inserted tube to measure the temperature at the center of the hydrogen peroxide solution. A 4mm. Pyrex rod was inserted into the solution from the mouth of the test tube to act as a stirrer. The test tube was insulated from the dry ice-acetone cooling bath by a concentric Pyrex test tube 16 mm. diameter. The resulting air gap was the limiting resistance to heat transfer so that the temperature read at the thermocouple was representative of the temperature throughout the stirred solution.

Thus far all of the experiments have been made by adding Pyrex glass particles of 38 to 42 microns diameter to the hydrogen peroxide solution.

The results obtained are summarized in the following table.

Table I

Freezing Points of 98-99% Hydrogen Peroxide to Which
38-42 Micron Pyrex Glass Particles Had Been Added

Run	Time Heated Prior to Addition of Pyrex	Observed F.P.	Wt. Pyrex Added (g).
4	4 min. at 42°C	-23°C.	.1003
5	4 min. at 42°C	-58°C	.1019
6	4 min. at 42°C	-28°C	.1032
7 a)	4 min. at 42°C	below -56°C	.0959
b)	- -	-53°C	--
8	4 min. at 42°C	-62°C	.2015
9 a)	15.75 min at 43°C	below -54°C	.0958
c)	- -	above -67°C	--
d)	- -	-64°C	--
10 e)	0 min.	below -58.5°C	.0998
b)	- -	-59°C	--
11 e)	0 min.	-33°C	.1069

Notes:

- a) First run of series.
- b) Second run of series performed 24 hrs. later.
- c) Second run of series performed 48 hrs. later.
- d) Third run of series performed 72 hrs. later.

It is apparent from the above results that considerable variations in the freezing temperature of the solution were possible. The cause of this variation is not apparent at this time.

In the next set of experiments seeding will be attempted

with 5 to 9 micron Pyrex particles. It is hoped to determine the effect of the particle size of the added substance on the freezing point of the hydrogen peroxide. Pyrex particles have been chosen for these initial experiments ^{because} the amorphous structure should have the least effect on the freezing point due to the surface orientation of any substance added for seeding. Other types of seeding materials, including crystals, are to be used in future experiments.

IV. High Pressure Ignition Limits of Hydrogen Peroxide Vapor (F. Feakes)

During the past report period, the equipment designed for the determination of the high pressure (1-10 atmospheres) ignition limits of hydrogen peroxide vapor has been constructed and partially assembled. It is anticipated that results will be available on the success of the apparatus at the time of next report.

The main features of the apparatus are as follows. Hydrogen peroxide vapor of a concentration approximating the estimated ignition limit will be generated in an electrically heated two liter Pyrex flask and superheated slightly (5-10°C) to prevent condensation. The vapor stream will be passed through an aluminum lined cylinder, which will act as the compression chamber, into an aluminum condenser from which condensate samples will be obtained for concentration determination.

A number of quick-operating plug and check valves have been arranged in the inlet and outlet streams of the cylinder in such a way that it should be possible to rapidly close both the inlet and outlet streams and compress the sample in the cylinder to a desired pressure. An aluminum faced piston with Teflon piston rings has been designed and it is believed that oxygen pressure will be a convenient method of activating the piston.

Immediately following the compression, the compressed vapor will be sparked between two fine platinum electrodes. The pressure during the compression and sparking will be recorded by a Brown Pressure recorder which has been adapted to record rapid pressure variations. Should ignition of the hydrogen peroxide occur there should be a noticeable rise in the pressure following sparking. In practice, it will be necessary to define a definite small pressure rise as indicating the occurrence of ignition at the limit. Pressure increases in excess of this will be judged as ignition while those of smaller magnitude will be considered non-ignition. The definition of this limit will depend on the sensitivity of the apparatus and the rate of decomposition of the hydrogen peroxide vapor in the test apparatus. The high rate of decomposition of hydrogen peroxide vapor has necessitated the use of glass, aluminum, and Teflon as the only materials available and suitable to be in contact with the vapor. Even so the rate of decomposition in contact with aluminum is so high that residence times in

the cylinder will have to be kept at a minimum. The initial tests should indicate whether or not it will be possible to compress, spark, and measure the pressure within such a time that the amount of decomposition will not be of significant amount.

V. Rate of Solution of Iron from Stainless Steel Immersed in Hydrogen Peroxide Solution (J. Chong)

Samples of three different types of stainless steel (302, 304 and 430) were immersed in 20 wt.% non-stabilized hydrogen peroxide (about 200 cc.) and the amount of iron dissolved in the solution (or present as a suspension) periodically measured. The size of the steel samples was 3 in. by 1 in. with a thickness of 20 gauge. The iron concentration in the hydrogen peroxide samples was determined colorimetrically: the peroxide was decomposed with acidic potassium permanganate solution and excess ammonium thiocyanate added. The optical density of the resultant color was measured with a calibrated model 400-A Lumetron Colorimeter.

The iron concentration in the hydrogen peroxide solution was found to increase within 5 to 10 hours to a maximum at a value of approximately 0.06 g. Fe/l. After this period the iron concentration in the solution appeared to decrease. After some 50 hr. of immersion the concentration then tended to level off to a constant value.

The results of tests with 304 and 430 types of stainless steel are presented in Fig. 3 and 4. The plots indicate that the extent of the concentration decrease following the maximum varied considerably.

A greater decrease was obtained in those runs where the sample was not pretreated with hydrogen peroxide and where the solution was allowed to stand in the bath unstirred. Also stirring appeared to limit the maximum concentration to a lower value--about 0.035 g Fe/l.

Stirring also had a considerable effect on the pH of the solution as shown in Fig. 4. On the assumption that hydrogen peroxide has no effect on the solubility product of ferric hydroxide, the maximum amount of iron present was of the order of 3×10^8 times that required for saturation at the prevailing pH, i.e. the iron was present presumably in some colloidal-suspended form. It is possible that the solution became supersaturated and the decreases following the maximum were the result of adsorption of the ferric hydroxide gel onto the walls of the Pyrex container and the stainless steel itself.

A preliminary test has indicated that about 15% of the concentration decrease of the iron is caused by adsorption onto the Pyrex. Further work is being planned to ascertain more definitely the cause of this concentration decrease.

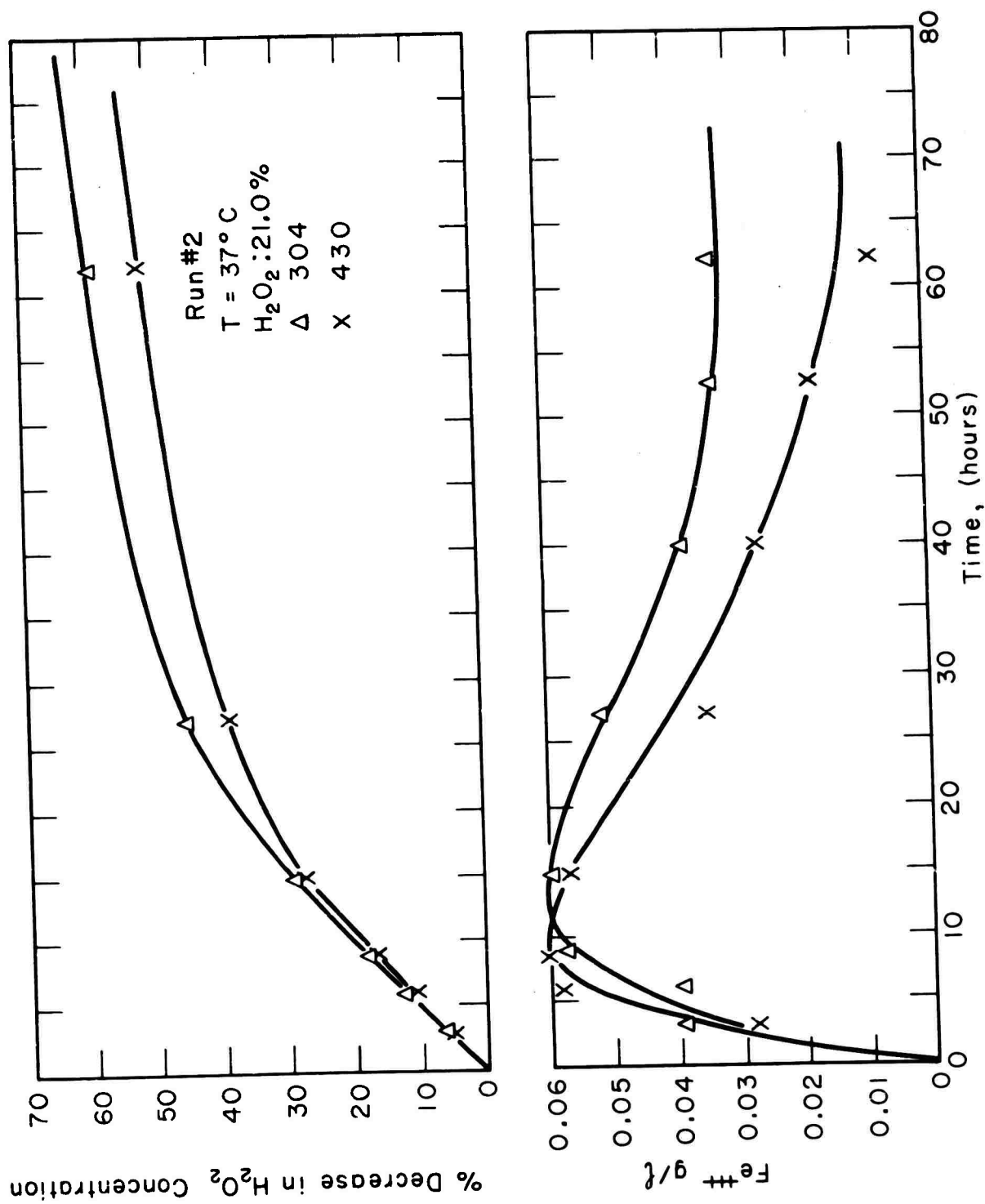


FIG 3 - SOLUTION OF IRON AND DECOMPOSITION OF H_2O_2 IN
 CONTACT WITH 304 AND 430 STAINLESS STEEL

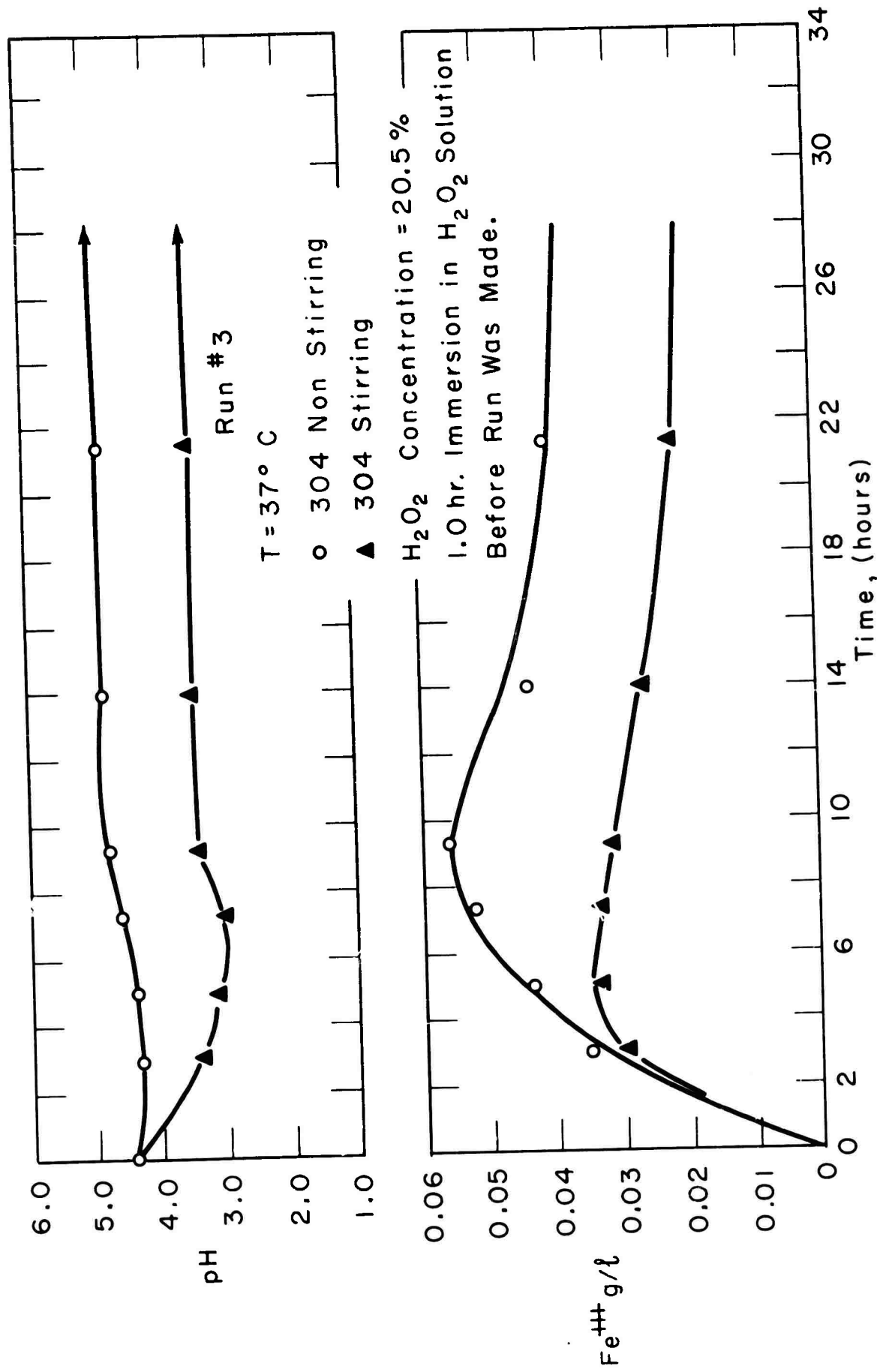


FIG 4- SOLUTION OF IRON AND ALTERATION OF pH IN H_2O_2 IN
 CONTACT WITH 304 STAINLESS STEEL

Experimental tests have indicated that manganese, potassium, and sulfate ions have no effect on the analytical procedure. According to the literature chromium and nickel are also supposed to be without effect.

VI. Literature Dealing with Hydrogen Peroxide

All the new material appearing in the Russian monograph mentioned in the last report has now been translated and the text is being edited preparatory to being distributed. The material added by the Russians to the basic German text does not include any novel material of importance. The Machu text has been taken as a framework and expanded here and there but the new material added is largely confined to scientific, polemic and review publications. There is no mention of military uses of H_2O_2 , although large sections of the new material deal with manufacturing techniques, handling and industrial uses. There is no mention of concerted Russian research on H_2O_2 per se and all material presented is abstracted from the literature, i.e. there is no original material reported in the book. On the other hand, the references quoted show awareness of German practice and of foreign patent, technical and engineering literature. A number of references quoted, both to Russian work or that of others, were new to us.

VII. Visitors

Mr. L. Powell, Office of Naval Research, Boston, Mass.
Mr. Donald Davenport, Stanford Research Institute

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